

Measurement of Photo-Polymerization Reaction (Methyl Methacrylate)

Polymethacrylate resin has been used in many plastic products and as an adhesive material. We have measured radicals which arise during the polymerization process of dental photo-curing methacrylate using ESR using the ultraviolet irradiation attachment (ES-USH500). The sample was placed in a sample tube and placed in the ESR cavity, irradiation was from the front window of the cavity. As shown in Fig. 1, immediately after the start of irradiation, a radical coming from the initiator was observed, and then a propagating radical derived from methyl methacrylate was observed with time. The spectrum, after the initiator radical disappears, is shown in the lower trace.

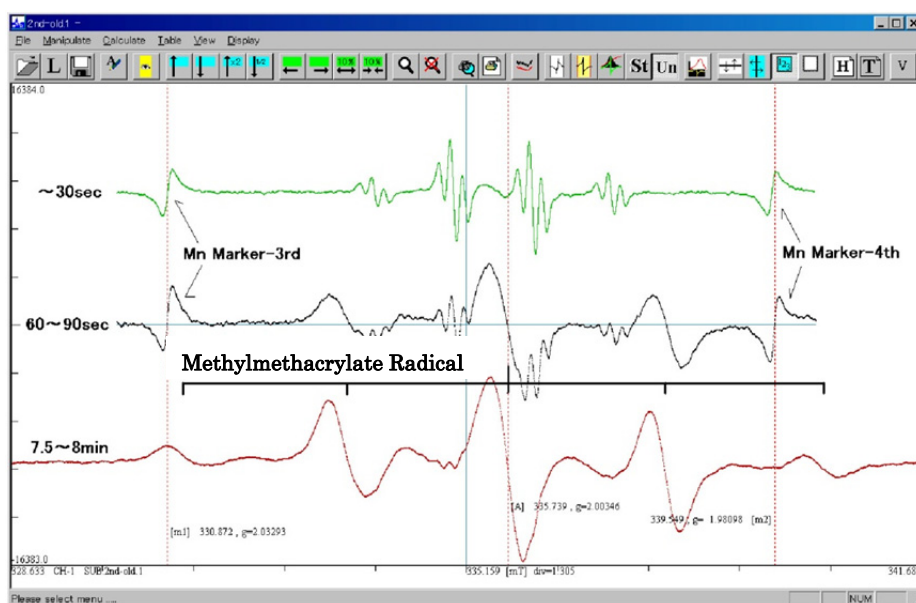


Fig. 1 Change of radicals in photo-irradiated methyl methacrylate

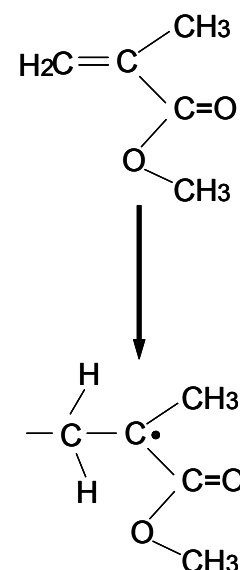


Fig.2 Methyl Methacrylate and the radical structure

In this radical, the three protons of the α -methyl group become equivalent to the unpaired electron by free rotation. However, as the rotation of the β -methylene group is restricted, one proton gives a coupling comparable to the α -methyl protons, whilst another proton does not show coupling with the unpaired electron (1). Therefore, the 4 protons of equal coupling, couple to the unpaired electron, and give rise to the spectrum of intensity ratio 1:4:6:4:1 as shown in Fig. 1. In such a reaction, polymerization proceeds further with time. The propagating radical arising during the process has been actively studied (2).

As can be seen from above, ESR is very useful for analysis of radical polymerization.

1 : T. Komatsu, T. Seguchi, H. Kashiwabara and J. Sohma *J.Polym.Sci.*, Part C. 16, 535(1967)

2 : A. Kajiwarra : *JEOL Ltd. News* 2008, pp 34-45

Sample: Courtesy of Tokuyama Dental Corp.